CHLORIDE IN SOILS

(An Arizona Method)

SCOPE

1. (a) This test method describes a procedure for determining chloride content in soil by a standard addition technique using a chloride electrode. The chloride content is defined in terms of the method and may be called water leachable or "available" chloride.

(b) This test method involves hazardous material, operations, and equipment. This test method does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

(c) See Appendix A1 of the Materials Testing Manual for information regarding the procedure to be used for rounding numbers to the required degree of accuracy.

(d) The extraction procedure, paragraphs 4 (a) through (d), is the same as is used in Arizona Test Method 733, Sulfate In Soils.

APPARATUS

2. Requirements for the frequency of equipment calibration and verification are found in Appendix A3 of the Materials Testing Manual. Apparatus shall consist of the following:

(a) 300 mL tall-form beaker (Pyrex #1060 or equivalent), calibrated to indicate 100 mL volume.

(b) An analytical balance capable of measuring the maximum weight to be determined and conforming to the requirements of AASHTO M231, except the readability and sensitivity of any balance utilized shall be at least 0.001 gram.

(c) Whatman #43 filter paper, or equivalent.

(d) Filtering funnel, 8 cm diameter.

(e) Magnetic stirrer and stirring bar.

(f) Erlenmeyer flask(s) - 500 mL capacity with stoppers.

(g) Chloride electrode system (Orion Ionplus #9617 combination chloride electrode or equivalent) suitable for use in the 300 mL beaker.

(h) Specific ion meter (Orion EA 940 or equivalent) suitable for use with the chloride electrode system.

(i) Thermometer, accurate to at least 0.5 °C.

(j) Pipettes of 1 mL, 5 mL, and 10 mL capacity, accurate to 1%.

(k) Sample bottle, 200 mL capacity or larger, with cap.

(l) Dropping bottle.

REAGENTS

3. (a) Chloride Standard Solution, 1.000 mg/mL (1000 ppm). (Fisher Scientific #LC13000-1, or equivalent, or made by transferring 1.648 gram dried primary standard sodium chloride into a 1 liter volumetric flask and diluting to 1000 mL with demineralized water).

(b) Nitric Acid, 20%. Measure 10 mL concentrated nitric acid into a clean beaker containing approximately 40 mL demineralized water and mix well. Transfer to a dropping bottle. **Caution must be exercised in preparing and using this solution! It should be properly labelled and treated as a hazardous material.**

PROCEDURE

4. (a) Weigh 100.0 ± 0.1 grams of soil passing a No. 10 sieve into a 500 mL Erlenmeyer flask.

(b) Weigh 300.0 ± 0.1 grams demineralized water into the flask.

(c) Stopper the flask, shake vigorously, and let the mixture stand one hour.

(d) Filter the extract into a sample bottle obtaining 125 to 150 mL of filtrate and put a cap on the bottle.

(e) Pipette a 10 mL aliquot of the filtrate into a 300 mL beaker. Record this volume as "A". Dilute to the 100 mL mark with demineralized water and add three drops of Nitric Acid, 20%. This is the "sample reading solution." Adjust the solution temperature to 25 ± 0.5 °C and maintain at that temperature throughout remainder of the test.

(f) Place a stirring bar into the beaker, place the beaker onto the magnetic stirrer, insert the electrodes, and initiate stirring. Stirring shall be at a constant moderate rate, such that the vortex created by stirring does not expose the tips of the immersed electrodes. The rate of stirring and the temperature (25 ± 0.5 °C) shall be constant throughout the procedure.

(g) After the reading has stabilized, record the initial reading, to the nearest millivolt, as "E_1".

(h) Add 1.00 mL Chloride Standard Solution.

(i) After the reading has stabilized, record the final reading, to the nearest millivolt, as "E_2".

(j) Calculate $\Delta E = E_1 - E_2$.

(k) If ΔE is less than 18, repeat paragraphs (e) through (j) with a smaller size aliquot.

(l) Prepare a reagent blank solution by placing 100 mL of demineralized water into a 300 mL beaker and adding 3 drops of Nitric Acid 20%. Repeat steps (f) through (j).

CALCULATIONS AND REPORT

5. (a) Calculate chloride concentration, "C_o" in the sample reading solution and "C_b" in the reagent blank reading solution, and record each to the nearest 0.010 mg/mL, as follows:

$$C_o \text{ or } C_b = \frac{1}{(101)\,10^{(\Delta E/S)} - 100}$$

Where: S = Electrode slope at 25 ± 1.0 °C as determined in accordance with manufacturer's recommendations. (The slope should equal approximately 59 millivolts for a properly functioning electrode.)

(b) Calculate chloride concentration in the soil in parts per million, "C", and report to the nearest 10 ppm as follows:

$$C = \frac{3 \times 10^5 (C_o - C_b)}{A}$$